

Reagents and conditions: (a) (i) SOCl₂, heat, 100%; (ii) CH₃OH, heat, 100%; (iii) Pd-C, CH₃OH, 100%; (iv) (i) NaNO₂, HCl, 0–5°C, (ii) KI, 91%; (v) (i) NaOH, THF-H₂O (3:1 v/v), (ii) eq. HCl, 93%; (vi) KBrO₃, 0.73 M H₂SO₄, 55°C, 3 h, 70%.

Fig. 1

Serial No: Not Yet Known Filed: Herewith
 For: USER- AND ECO-FRIENDLY HYPERVALENT IODINE
 REAGENT AND METHOD OF SYNTHESIS
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Entry	Substrate	Product	Conditions ^a	% Yield
1			1:2, H ₂ O, RT, 18 h	86 ^b
2			1:2, H ₂ O, RT, 18 h	80 ^b
3			1:2, H ₂ O, RT, 18 h	80
4			1:1.5, H ₂ O, 60°C, 3 h	85
5			1:1.5, H ₂ O:THF (5:2 v/v) ^c 60°C, 3 h	79
6			1:1.5, H ₂ O:THF (5:2 v/v) ^c 60°C, 3 h	89
7			1:1.5, H ₂ O, 60°C, 3 h	85
8			1:2.5, H ₂ O, 60°C, 3 h	80
9			1:1.5, H ₂ O:THF (4:3 v/v) ^c 60°C, 3 h	86
10			1:2.5, H ₂ O:THF (3:1 v/v) ^c 60°C, 3 h	81
11			1:1, H ₂ O, 60°C, 3 h	80 ^d
12			1:2 H ₂ O, 60°C, 3 h	84

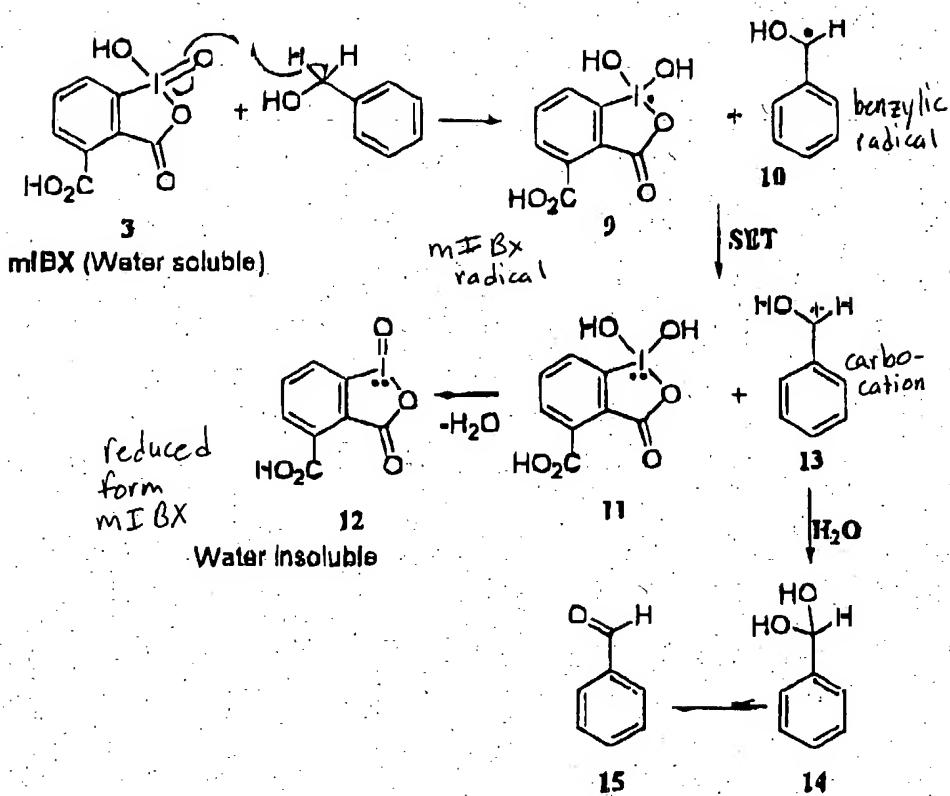
^a Reaction conditions are noted in the following order: Substrate to mIBX ratio, solvent(s), temperature, time

^b The yield reported for entries 1 and 2 are calculated using gas chromatography. The other yields reported in Table 1 are isolated yields.

^c Formation of γ-butyrolactone (~10%), via oxidation of THF, was observed from reactions carried out in the mixed solvent system.

^d Nearly 25% of phenylglyoxalic acid was also isolated from this reaction.

FIG. 2



Mechanism of oxidation of benzylic/allylic alcohols in water using mIBX.

FIG. 3